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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application of
DENNIS MAPLES et al



) July 17, 2000

Serial No.: 09/419,856) Group Art Unit: 1711

Filed: October 19, 2000)

For: LOW SMOKE, LOW TOXICITY) Examiner: U. K. Rajguru
CARPET) (703) 308-3224

DECLARATION UNDER 37 CFR 1.132 OF TIMOTHY W. JOHNSON

I, Timothy W. Johnson, hereby declare and state:

1. I am a 1963 graduate of Trinity College (Hartford, Connecticut), where I received the degree of Bachelor of Science in Chemistry. I further obtained a Master of Science degree in Physical Chemistry at Trinity College in 1965. I then worked briefly for Loctite Corporation on development of adhesives and sealants. Subsequently I obtained a Ph.D. in Physical Chemistry from Purdue University in 1970. From November, 1970 to July, 1973 I worked at Northwestern University in Evanston, Illinois as a visiting research scholar in the field of water-soluble polymers. Subsequently I worked from August, 1973 to January, 1999 in polymer research and development at Phillips Petroleum Company in Bartlesville, Oklahoma. Since February, 2000, I have been an employee of Akro Fireguard Products Company ("Akro"), the assignee of the above-identified application, where I am working primarily in development of fire-resistant materials and structures.

2. Specifically, over the last 30-plus years, I have worked in polymer science including synthesis and characterization of polymeric materials including polyolefins, engineering plastics, and composite materials. Specific polymers with which I have worked include polyethylene and ethylene-olefin copolymers, polypropylene, butadiene styrene copolymers, polyphenylene sulfide, polymer thickeners for motor oil, vinyl imidazole-vinyl pyrrolidone copolymers, polyethylenimine and acrylic polymers. Specific methodologies that I have used in my work include nuclear magnetic resonance spectroscopy, viscometry, rheometry, thermodynamics, kinetics, differential scanning calorimetry, thermogravimetric analysis, and gel permeation chromatography. I have 17 publications and 23 U. S. patents as a result of my professional work.

3. I am familiar with the disclosure of this application and I am also familiar with the Office Action mailed February 17, 2000, and the rejection based on the Scholz patent 5,749,948.

4. The following experiments were carried out under my direction and control to compare the composition of Example 9 of Scholz with the composition according to the above-identified application.

Run 1 - Composition According to Present Invention

5. I found the following preparation from the notebook of one of the inventors, Dennis Maples, and it illustrates a composition according to the above-identified application. I

report it here for comparison. It is an example of a carpet backing formulation, which, when applied to a carpet forming material, provides a good smoke rating.

#	Component	Amount (%)	Source
1	PR-240	22.4	Bayer
2	Water	3.9	
3	2-propanol	.42	
4	N-methylpyrrolidone	.76	
5	CT-131	4.0	Air Products
6	Melamine	10.4	
7	FR CROS 484 ammonium polyphosphate	31.4	Budenheim
8	Pentaerythritol	10.4	
9	Black Pigment 5099	1.1	
10	Silica	2.3	
11	DF 574	0.06	
12	PR-240	11.4	Bayer
13	Surfynol 420	0.23	Air Products
14	Surfynol 465	0.57	Air Products

The following note describes the changes by category (see original claim 11 for nomenclature) in ingredients from those used in the original formulation when the parent application was filed on April 15, 1998.

1. filler, pigment, anti-settling and grinding aid

Silica was added in the small amount of 2.3%. I have learned that the silica was Min-U-Sil 3, a powdered silica. I

determined by inspection of a bottle of it that it is a high density, low surface area (as contrasted with fumed silica) powder. Thus it is present as an (non-flammable) inert ingredient.

2. surfactants

Surfynol 420 is a surfactant made by Air Products used in a very small amount of 0.23%. Surfynol 420 is an ethoxylated 2,4,7,9-tetramethyl-5-decyn-4,7-diol. As such it is similar to Surfynol 465, also used in this example. The two surfactants differ in the amount of ethoxylation. Both are designed to lower the surface tension of the aqueous medium that is part of the material.

The above ingredients were mixed in order with strong stirring. Subsequently the mixture was diluted with 63 grams of a mixed solvent consisting of water (76.7%), N-methylpyrrolidone (15.0%) and 2-propanol (8.3%).

The mixture was then coated onto a nylon carpet at a rate of 25 oz/yd², dried, and tested for smoke generation according to FAR 25.853(d), Appendix F, Part V. In this test an area of sample material is subjected to high radiant intensity from an electric heater with provision for ignition of off-gases, for a period of 4 minutes. Smoke generated from this treatment is retained in a compartment that encloses the sample and heating and ignition means. The optical density of the enclosed space is monitored during the course of the four minutes, and the optical density is recorded at the end of the four minutes. The

resulting number gives the smoke parameter according to the test. The test results on three specimens were 181, 115 and 99, average 132.

In the Akro application and this declaration there are references to several fire and smoke tests and criteria, from sources including Boeing, Airbus and the Federal Aviation Administration. The test methods are all derived ultimately from the NBS (National Bureau of Standards, now NIST) smoke chamber test. Boeing standard 7238 refers directly to AMINCO-NBS Smoke Density Chamber, Cat. No. 4-5800B, instruction 941-B dated May, 1983. Airbus and Federal Aviation Regulations both refer to ASTM F814. ASTM F814, in turn, refers to the NBS smoke chamber. Akro's independent testing expert, Mr. Herb Curry, Mt. Vernon, IN 47620 (Phone: 812-838-6703) considers the smoke test methods as the same. In a recent conversation he encouraged our use of the designation FAR 25.853(d), Appendix F, Part V. I have adopted this designation for the smoke test in this declaration.

Each of the three sources recognizes the importance of low smoke performance. They have different criteria: Airbus specifies 150 and Boeing specifies 300. Akro personnel have talked with Airbus and Boeing personnel responsible for approving and purchasing on multiple occasions regarding these specifications. They have learned that the two companies view the specifications in different ways. Boeing personnel have indicated that they would not approve a carpet with a rating of 300, and, when approached by Akro with a carpet with a smoke

rating of 220, they suggested that we work to achieve lower smoke levels. In contrast, Airbus personnel have indicated that the 150 specification is rather strict, and that they would be willing to consider recommending carpets that approach a 150 smoke rating even if they do not meet it. Based on these experiences Akro management and technical staff believe that a smoke rating of 200 will be needed and will be sufficient to meet airline needs; we have thus made it our target maximum. This criterion of 200 coincides with the criterion in FAR 25.853(d), Appendix F, Part V for aircraft compartment interiors.

The examples in this declaration demonstrate that the Akro inventive carpet backing has a smoke rating below 200, and that the Scholz formulation, when modified as needed to make it applicable as carpet backing, exceeds the 200 rating by a significant margin.

Run 2 - Scholz - First Attempt

6. The initial attempt was to make the composition of Example 9 of the Scholz patent by adding the materials in the order of the list in the example. During the course of the addition the materials the resulting mixture became too thick to stir. I concluded that the order of addition implied in Example 9 is simply a list of ingredients, and not a specification of order of addition. The order of addition which was subsequently used is that consistent with best experience and practices known to Akro and others.

Run 3 - Scholz - Better Order of Addition

7. The Scholz composition was made with a better order of addition.

	Composition	Amount (%)	Source	Comments
1	Impranil DLP	20.8	Bayer	
2	Water	20.5		
3	Lopon 890	0.2	BK-Giulini	
4	Titanium dioxide	4.0	Kemira	
5	Pentaerythritol	12	Hoechst-Celanese	
6	Ammonium polyphosphate, AP 462	24.0	Hoechst-Celanese, now Clariant	Epoxy-coated ammonium polyphosphate
7	Melamine	14.0	The Melamine Co.	
8	Ethanolammonium salt of mixed mono- and di- ethyl esters of phosphoric acid	1.5	Prepared at Akro. See below for details.	
9	Tylose C-30	3.0	BK-Giulini	Carboxymethyl- cellulose

To make the monoethanolammonium salt of Knapsack phosphate MDE, I attempted to first obtain Knapsack phosphate MDE. This Knapsack phosphate MDE was previously made by Hoechst. According to a personal communication from Clariant, current suppliers of this material, under the trade name Hordaphos MDE, it is a mixture of mono and diethyl esters of phosphoric acid. I was unable to obtain Hordaphos MDE at the time I needed it. Thus I prepared a "Hordaphos equivalent" by reaction of ethanol with phosphorus pentoxide. I then prepared the ethanolammonium salt by reacting my Hordaphos equivalent with ethanolammine.

Components 1 through 3 were combined and mixed. Subsequently components 4 through 7 were added sequentially, slowly, with high speed stirring using a dissolver blade, periodically scraping the sides and bottom of the vessel. After these additions were complete, the mixture was stirred at high speed for an additional 10 minutes with the dissolver blade. During this mixing process, the mixture became thick, and warmed up as a result of the mixing energy. It was cooled to room temperature, and component 8 was added and mixed. The mixture was still very thick or stiff. Component 9 was added, and the mixture was mixed, first with a paddle and then with a dissolver blade. The resulting mixture was very thick, in fact a rubbery semisolid, resistant to permanent deformation. In view of its resistance to deformation as needed for spreading, I did not attempt to apply it to carpet.

Run 4 - Scholz with Added Grind Aid

8. I noted that the Scholz composition does not contain a "grind aid". A grind aid is a material which facilitates break-up of aggregates and agglomerates of particles of the active ingredients such as ammonium polyphosphate, melamine, titanium dioxide, and (di)pentaerythritol. The particulate ingredients need to be fine to penetrate into a carpet fiber structure. I prepared a batch according to the Scholz Example 9, with the substitution of Air Products CT-131 grind aid for a similar weight of water as in Run 3 to increase the flow.

#	Components	Amount (%)	Source
1	Impranil DLP	20.8	Bayer
2	Water	20.2	
3	Lopon 890	0.2	BK-Giulini
4	CT-131	0.3	Air Products
5	Titanium dioxide	4.0	Kemira
6	Pentaerythritol	12.0	Hoechst- Celanese
7	Ammonium polyphosphate AP462	24.0	Hoechst (now Clariant)
8	Melamine	14.0	The Melamine Co.

9 Ethanolammonium
salt of mixed mono- 1.5 Prepared at
and di-ethyl ester
of phosphoric acid Akro. See Run
 3 for details.

10 Tylose C-30 3.0 BK-Giulini

Components 1 through 4 were combined and mixed. Subsequently components 5 through 8 were added sequentially, slowly, with high speed stirring using a dissolver disk, periodically scraping the sides and bottom of the vessel. After these additions were complete, the mixture was stirred at high speed for an additional 10 minutes with the dissolver disk. At the end of this time the mixture was still fluid. This constitutes a difference relative to the previous example, Run 3, without a grind aid, which was viscous to the point of being non-flowable. Component 9 was added, and the mixture was again stirred. The viscosity did not change noticeably. Finally the Tylose was added over a 15 second period. Mixing was continued for an additional 5-10 minutes, with difficulty as a result of the thickening effect of the Tylose.

The resulting material was spread onto and driven into velvet carpet at a rate which gave about 28 oz/yd² after drying. The coating material did not penetrate well into the carpet as a result of its thick, semisolid texture. The physical properties of the resulting carpet were noted. The carpet was stiff as a

result of the backing. When the carpet was flexed, the backing cracked. When the front (top) of the carpet was tested by rolling with a Velcro® roller, substantial fuzzing was observed, indicating that the fibers had not been bound in place by the backing. Thus the composition is unacceptable as a carpet backing. Velvet carpet is the easiest carpet for a backing material to penetrate, in Akro experience. It is substantially easier to penetrate velvet carpet than others that Akro is called upon to treat. Since this example coating material does not satisfactorily treat velvet carpet, it is reasonable to expect that it will also not satisfactorily treat the other carpets that are presented for treatment.

Run 5 - Scholz with Addition of Grind Aid and a Small Amount of Extra Water

9. The Scholz composition was made with the addition of a grind aid and a small amount of extra water.

	Component	Amount (%)	Source
1	Impranil DLP	20.0	Bayer
2	Water	15.2	
3	Lopon 890	0.2	BK-Giulini
4	CT-131	0.3	Air Products
5	Pentaerythritol	11.0	Hoechst Celanese

6	Ammonium polyphosphate, 22.1 AP 462		Hoechst (now Clariant)
7	Melamine	13.2	The Melamine Co.
8	Titanium dioxide	3.7	Kemira
9	Water	15.0	
10	Ethanolammonium salt of 1.5 mixed mono- and di- esters of phosphoric acid		Synthesized at Akro. See Run 3 for details
11	Tylose C-30	3.0	BK-Giulini

Components 1 through 4 were combined and mixed. Thereupon components 5 through 8 were added sequentially, gradually with high speed stirring with a dissolver blade. Addition was at such a rate that the powder was blended in as it was added. High speed stirring was continued for 10 minutes after addition of components 5 through 8 was completed. Thereupon components 9 and 10 were added, and mixing was continued. Thereupon the speed of the stirrer was reduced. Component 11 was added with stirring over a period of 15 seconds, to allow dispersion of the particles before they started to

swell. Stirring speed continued at low speed for 5 minutes.

After this preparation, the consistency of the material was similar to cake frosting.

A 200g portion of the coating described above was applied to the back side of about a 12 x 16 inch area of velvet carpet. The material was spread with a 5-inch wide trowel in such a manner as to spread the material as uniformly as possible over the designated area, and then to drive the backing into the carpet as deeply as possible, given the resistance to flow of this material. Thereupon the coated carpet was placed coated-side-up in an oven at 270°F for 30 minutes. The carpet was then removed from the oven and allowed to cool to room temperature. Thereupon an additional 200g portion was applied to the back of the carpet, on top of the previously coating. This two coating step procedure was used as employed by Scholz in his Example 1 from which the other examples follow. As previously, the coating was spread with a 5-inch wide trowel as uniformly as possible, and pressed into the previous coating to the limited extent possible. Thereupon the coated carpet was placed coated-side-up in an oven at 270°F for 30 minutes. The carpet was then removed from the oven and allowed to cool to room temperature.

The carpet was stiff. When a portion of it was flexed, the backing cracked. The front (top) of the carpet was tested by rolling with a Velcro® roller to look for signs of fuzzing, which indicates that the coating has not bound the fibers in place. There was an unacceptably high level of fuzzing. This is due to

the fact that coating did not penetrate into the fiber bundles enough to bind the fibers, which, in turn, is due to the fact that the coating mixture has a high resistance to flow. A portion of the coated carpet was sampled for smoke testing according to FAR 25.853(d), Appendix F, Part V for 4 minutes by an independent test laboratory (Herb Curry). The smoke rating was 307 after 4 minutes. As discussed in Section 5 above, this does not meet the industry target maximum of 200 nor the criterion in FAR 25.853(d), Appendix F, Part V for aircraft compartment interiors.

Run 6 - Scholz - Made Fluid with Addition of Grind Aid, Substantial Addition of Water, and Elimination of Tylose Thickener

10. Previous attempts to make a functional coating useful as a carpet backing based on Scholz have resulted in materials that resist flow to the extent that they do not penetrate the carpet fibers enough to bind the fibers and prevent fuzzing under simulated wear conditions, and, when applied at the levels specified by Scholz, give a carpet which is stiff, the backing of which cracks under moderate flexing. In the previous example, Run 5, the resulting carpet was also shown to give unacceptably high levels of smoke when subjected to a standard test.

The present example attempts to relieve the flow restriction by addition of more water and elimination of Tylose thickener. The components and amounts are as follows:

	Component	Amount (%)	Source
1	Impranil DLP	10	Bayer
2	Lopon 890	0.2	BK-Giulini
3	CT-131	0.3	Air Products
4	Water	19	
5	Pentaerythritol	11.4	Hoechst- Celanese
6	Ammonium polyphosphate, AP 462	22.9	Hoechst (now Clariant)
7	Melamine	13.3	The Melamine Co.
8	Titanium dioxide	3.8	Kemira
9	Impranil DLP	10.5	Bayer
10	Water	7.0	
11	Ethanolammonium salt of mixed mono- and di- ethyl esters of phosphoric acid	1.5	Synthesized at Akro. See Run 3 for details.

Components 1 through 4 were combined and mixed. Thereupon components 5 through 8 were added sequentially, gradually, with high speed stirring with a dissolver blade. Addition was at such a rate that the powder was blended in as it was added. High speed stirring was continued for 10 minutes after addition of components 5 through 8 was completed. Thereupon components 9 and 10 were added, and mixing was

continued. Thereupon components 9 through 11 were added, and stirring was continued. The mixture, which was warm as a result of stirring, was cooled to 19°C and the viscosity was measured with a Brookfield viscometer (spindle #3): 9780 cP @ 20 RPM and 37400 cP @ 2 RPM. This viscosity is still too high. Thus 5% additional water was added in two increments, bringing the viscosity to 4880 cP @ 20 RPM and 17600 cP at 2 RPM.

The coating was applied to velvet carpet at 28 oz/yd² (weight after drying). A 5-inch trowel was used to apply the coating, in the first instance to spread it uniformly over the carpet, and in the second to force it into the fiber structure. It was apparent to the eye that the coating worked into the fibrous structure. After drying at 270°F for 30 minutes, the carpet was allowed to cool to room temperature. The resulting carpet was flexible, and, when flexed there was no sign of cracking of the backing material. A portion of the carpet was tested for fuzzing under the affect of the Velcro® roller. There was, at most, a small amount of fuzzing. A second portion of the coated carpet was samples for smoke testing according to FAR 25.853(d), Appendix F, Part V for 4 minutes by an independent test laboratory (Herb Curry). The smoke rating was 257 after 4 minutes. As discussed in Section 5 above, this does not meet the industry target maximum of 200 nor the criterion in FAR 25.853(d), Appendix F, Part V for aircraft compartment interiors.

This example shows that when the Scholz composition is modified so that it can be effectively driven into the carpet,

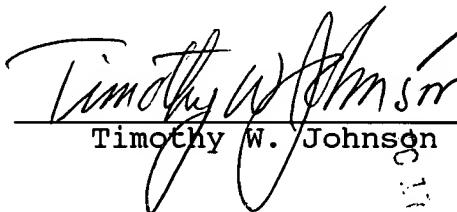


more time and energy is required in the drying process.

Furthermore, the carpet still does not pass the smoke test.

11. I hereby declare that the foregoing statements are made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further that these statements were made with knowledge that willful false statements and the like so made are punishable by fine or imprisonment or both, under § 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent to issue therefrom.

July 17, 2000


Timothy W. Johnson

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